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PROTOCOL FOR SAMPLING AND EVALUATING FLY ASH FROM NON-HAZARDOUS SOLID WASTE INCINERATION FACILITIES

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Ontario

Ministry of
Environment
and Energy

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PROTOCOL

FOR SAMPLING AND EVALUATING FLY ASH

FROM

NON-HAZARDOUS SOLID WASTE

INCINERATION FACILITIES

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INTRODUCTION:

Concerns have been raised throughout North America and Europe about the leachate toxicity of fly ash (i.e. fly, boiler, economizer, baghouse dust, etc.) from energy from waste plants (EFW) with respect to heavy metals. A Federal program, in which the Ontario Ministry of Environment participated [National Incineration Testing and Evaluation Program (NITEP)], investigated the problem and found that in most cases untreated fly ash failed the Ontario Regulation 309 leachate test. Bottom ash rarely failed the test.

The following report contains Ontario guidelines for municipal solid waste incinerator ash handling and fly ash sampling resulting from the incineration of waste that is neither hazardous waste nor liquid industrial waste. It has been prepared for guidance to Approval engineers in the issuance of a Certificate of Approval for an incineration facility and also for the owners of such a facility so that they may see what is expected of them in handling ash and sampling fly ash.

The Approvals Director of the Ministry of the Environment (hereafter referred to as the Director) may at his/her discretion require a change in the method of analysis, sample frequency or test requirements.

1) ASH HANDLING:

- Fly ash from the incineration of municipal solid waste (MSW), effective September 1, 1990, will no longer be exempted from being hazardous waste under Ontario Regulation 309 1. (27 XV) due to amendment, Reg 138/90. Fly ash will therefore have to be assessed prior to disposal for leachate toxicity and other Regulation 309 hazardous characteristics to determine disposal alternatives.

- Combining and disposing of hazardous fly ashes with bottom ash is no longer permitted. Non-hazardous fly ash can be disposed in non-hazardous disposal areas which are approved for handling such material.

- To minimize fugitive emissions, fly ash may be transported within the plant pneumatically or must be in enclosed mechanical conveyors unless the ash is handled as a slurry (i.e. predominantly water). Safety aspects of dry ash handling should be considered (e.g. fire/explosion).

- All ash collection, storage and transfer from conveyors to containers, must be carried out indoors and be designed to minimize fugitive emissions. Totally enclosed storage silos for fly ash may be located outside of the building. Any vents fitted to the silo must have vent filters which should be inspected monthly.

- Drainage from ash storage piles within the plant must be collected and may be reused within the plant or be discharged with appropriate treatment to meet effluent criteria.

- Transportation of ash must be by a properly licensed carrier.

- Solidified/stabilized ash in block form may be transported dry to an appropriate landfill and does not need to be covered during transportation. All other ash being transported to a hazardous or non-hazardous landfill must be wet (or damp) and enclosed in a suitable container to prevent fugitive emissions. (However, it must still pass the slump test if going to a non-hazardous landfill.)

- Fly ash containers may not be used for any other purpose unless thoroughly cleaned prior to the other use. (Note: A truck body is considered to be a container.)

- Doors or tailgates must be gasketed to prevent any water contained in the ash from being spilled on the road.

- Ash being deposited in an appropriate landfill must be handled with care during spreading and compaction and must be covered or kept damp to minimize fugitive emissions.

- The owner/operator shall keep records of the tonnage of ash being disposed and the final destination of the ash.

2) FLY ASH SAMPLING:

Fly Ash shall be assumed to be leachate toxic unless the owner can prove that the fly ash is not hazardous.

The purpose of sampling fly ash in a prescribed manner is to obtain representative samples that can be analyzed to determine if the fly ash requires management as a hazardous waste as defined in Regulation 309.

Ash sampling programs and proposed methods must have prior approval of the Director.

All facilities shall be designed to provide easy and safe access for the sampling of all fly ash or commingled fly ash streams. The owner or operator shall provide a plan to the Director showing the location and access of each sampling port, along with proposed methodology of sample collection. Any sampling equipment required shall be provided by the owner or operator and be kept in clean and good condition.

Sampling shall be conducted by the owner or operator or by their designate.

The owner or operator shall give Fourteen (14) days advance notice before each sampling period, to the local Ministry Office so that auditing the collection of the samples may be arranged.

Also, the owner or operator shall provide daily records of all plant operating conditions while sampling is taking place.

The records shall include but may not be limited to:

- characteristics/type of waste processed;
- tonnage of waste processed;
- temperature of primary and secondary chambers*;
- oxygen level at the exit of the secondary*;
- carbon monoxide levels* and opacity* at exit of the pollution control system or "in stack"; and,
- tonnage of fly ash transported from the plant.

* Hourly, numerical maximum, minimum, mean & 8 Hour graphical trends.

- A clean secure area shall be provided for sample storage during the sampling period. This need not be refrigerated provided that ice packs are readily available when required by the nature of the sample, i.e. samples taken for organic analysis.

3) **FREQUENCY OF TESTING:**

Testing shall consist of quarterly five-day test periods spaced evenly throughout the year. This shall be done for every year that the plant is in operation unless otherwise approved in writing by the Director or the fly ash is sent to a hazardous waste facility.

For each of the five days, a 24 hour composite of fly ash shall be submitted for analysis. The leachate extraction procedure (LEP) (see Ontario Regulation 309) shall be conducted in triple replicate on each sample for the metals listed in Table 1.

Analyses for PCB and Table 2 organics shall be performed on one sample from the first and second sample periods only, unless otherwise required by the Director.

4) INTERPRETATION OF RESULTS

The above procedure will produce 15 sets of data for metals per quarter for fly ash. Statistical analysis of the data will be used to determine if the fly ash is leachate toxic.

The following programs are based on statistical methods found in "Experimental Statistics, Handbook 91". Published by the National Bureau of Standards, United States department of Commerce. The methods have been adapted to Lotus 1-2-3 (Version 2.01) and were run on an IBM AT clone.

Statistical Test

The first program "Statistical Test for Incinerator Ash Leachate Toxicity" (see Appendix) asks for the parameter of concern (e.g. Lead, Cadmium etc.) and the leachate quality criteria value. (i.e. the concentration from Schedule 4, Reg. 309) The analytical data are then entered (maximum of 19 data points) (if less than 19, keystroke [ENTER] until matrix is filled). The program then calculates the degrees of freedom and calls the "student t" value for n-1 degrees of freedom, single tailed 90% confidence level into the equation. The program calculates standard deviation (s) of the samples and then places the values in the equation U = t.s/sqrt(n)
The value produced is then added to the true mean (x) and if this is less than the regulatory value the waste is considered non-hazardous. If the true mean plus the value produced is greater than the regulatory value the waste is judged to be leachate toxic.

Outliers

Data may contain one point which is numerically different (an outlier) from the rest. A decision needs to be made on whether to keep the suspect data point in the computations, whether to discard it as being a faulty measurement or whether to give the measurement a diminished status in the final outcome rather than the two extremes i.e. full vote - diminished vote - no vote.

It has been decided that it is better to exclude the data point, in that when a good measurement (i.e. valid) is excluded, we simply lose some of the relevant information with a decrease in precision and the introduction of some bias (both theoretically calculable) while if a truly anomalous measurement is included it invalidates the results, biasing both the final average and the estimate of precision by unknown and generally indeterminate amounts.

If, on inspection of the analytical results of the ash, a single value appears to be inconsistant, i.e. an outlier, the second program "Statistical Test for Outlier Rejection", (see Appendix) should be used to determine if the value can be rejected. The program again asks for the parameter of concern and the value from Schedule 4. All of the data are then entered and again [ENTER] is keystroked until the matrix is filled. The program used is the "Dixon Criterion for Outliers" which is the method recommended by the United States Environmental Protection Agency (See SW846). The Printout of results will indicate if the high value should be rejected. (N.B. a minimum of 10 sample values are required to perform this test)

If the high value should be rejected only the remaining values should be run through the "Statistical Test for Incinerator Ash Leachate Toxicity" i.e. program 1.

Application of Results

A copy of the spread sheet program used for the interpretation of data on a statistical basis and a spread sheet program to test for outliers is provided in the Appendix.

NOTE: Use of trade names does not constitute a commercial endorsement.

If the fly ash, based on the analysis of the samples from two consecutive quarterly test periods is not found to be statistically leachate toxic, and is not hazardous by other criteria, it can be disposed of at an approved municipal landfill site without further treatment. If the results from one quarters test period indicate a leachate toxic material, then the fly ash will need to be treated or sent to an approved hazardous waste landfill.

5) SAMPLING METHODOLOGY & PREPARATION:

The preferred method of sampling is to collect all the fly ash produced per eight-hour shift, in drums and then sample with a tube sampler. An equal number of full depth tube cores should be taken from each drum to produce approximately eight litres of ash per shift. Alternatively a composite sample shall consist of hourly, equally sized, grab samples.

The three shift samples should then be combined, thoroughly mixed, coned and quartered on a steel plate or smooth clean concrete floor. [Note: The use of a sample splitting (riffle) device is not recommended for sample size reduction of this material]. Two diagonally opposite quarters should be selected on a random basis, remixed, coned and quartered until the sample has been reduced to approximately 1 litre. The last set of discarded quarters may be archived as back up samples until analysis is complete.

Samples for Metal Analyses:

The samples should be placed in clean glass or P.E.T. (Polyethylene Terephthalate) 1 litre wide mouth jars fitted with a lined lid. Type of waste sampled, field sample number, sampling location, time, date and sampler's initials should be marked on the label. The bottle should then be stored in a secure area to comply with "chain of custody" requirements. Appropriate "Request for analysis" sheets, duly filled out and signed by the sampler, must accompany the sample.

Samples for Organic Analyses:

The sample taken for the determination of organics shall be placed in a proofed (i.e. certified clean by the analysing laboratory) glass jar, capped immediately and sealed with non-adhesive Poly Tetra Fluro Ethylene (PTFE) pipe thread tape. The Jar Cap liner shall be of PTFE or other approved material. The sample should be kept below 5°C during transport and storage to inhibit the loss of volatile components. Type of waste sampled, field sample number etc. should be marked on the label similarly to the sample for metal analysis.

Field Record of Sampler:

The field record should be kept for 2 years and contain the number, time, date and nature of each sample taken, the name of the operator or designate who took the sample and the name of the person who supervised the sampling.

Analysis:

All analytical methods used, shall be in conformity with standard methods contained in the 'Handbook of Analytical Methods for Environmental Samples', Ontario Ministry of the Environment, Laboratory Services Branch (LSB). Deviation from the above must have prior approval of the Director.

The Leachate Extraction Procedure (Reg. 309) is to be done in triplicate on each day's sample of fly ash for the following schedule 4 metals in Table 1.

TABLE 1: METALS OF CONCERN FOR LEACHATE TOXICITY IN FLY ASH

(Schedule 4)

Arsenic	Lead
Barium	Mercury
Boron	Selenium
Cadmium	Silver
Chromium	

Leachate Extraction Procedure (LEP) analysis for PCB is to be done on one 24 hour composite (Day 4) of the first and second set of the first year's samples.

Analysis for the following Regulation 309, **Schedule** 3, severely toxic contaminants is to be done on the original fly ash (not on the leachate from the LEP). One 24 hour composite (Day 4) of the first and second set of the first years samples is to be analysed.

TABLE 2: SEVERELY TOXIC CONTAMINANTS THAT MAY BE PRESENT IN FLY ASH

(Schedule 3 Organics)

2,3,7,8-Tetrachlorodibenzo-p-dioxin
1,2,3,7,8-Pentachlorodibenzo-p-dioxin
1,2,3,4,7,8-Hexachlorodibenzo-p-dioxin
1,2,3,6,7,8-Hexachlorodibenzo-p-dioxin
1,2,3,7,8,9-Hexachlorodibenzo-p-dioxin
2,3,7,8-Tetrachlorodibenzo furan

The results of all analyses are to be reported to the local District Officer of the Ministry of the Environment.

Audit Samples

In addition to the foregoing sampling, the Ministry may, at its own discretion, conduct unannounced sampling of the ash materials. The owner or operator shall provide records of plant conditions when requested. The owner or operator shall also provide all help, guidance and special equipment required in order to take the samples safely.

6) SAFETY DURING SAMPLING

Sampling fly ash presents some health and safety concerns. The material contains toxic metals and organics, is usually hot when collected and may contain unreacted lime. Because of this, it is advisable not to inhale the ash or get it on bare skin or in the eyes.

The following procedures and precautions are offered as suggestions and are based on practical experience and observations gained from testing programs carried out at a number of MSW incineration facilities. This does not relieve the owner/operator from strict adherence to legislated occupational health and safety requirements (including WHMIS) and to any other in-plant safety procedures.

Dust masks, safety goggles or full face shield, clean coveralls (changed daily) and gloves are highly recommended. The use of barrier creams is not recommended as they prolong contact with the ash that might otherwise fall off the skin. (It is assumed that the plant requires the mandatory use of approved steel toed footwear, hardhats and safety glasses).

Sampling personnel who may be unfamiliar with the plant are cautioned to take note of the incineration process going on around them and the physical layout of their immediate area, including emergency exits. They are also cautioned to observe plant rules such as lock out procedures or confined space entry procedures. They are specifically cautioned not to stand under the rams located in the primary chamber of starved air incinerators, as red hot ash occasionally sticks to the ram on the withdrawal stroke.

They are cautioned against getting too close to the primary quench tank when bottom ash is being transferred from the primary chamber. A load of bottom ash, high in metals content, can cause local boiling in the tank with a real danger of scalding.

Another area to be avoided if at all possible, is the inspection doors of the primary chamber when waste is being charged to the incinerator. If the load contains highly volatile materials (e.g. plastics, or a can of solvent) the chamber may become slightly pressurized and flamable gas or flame can leak around door edges.

Damp or wet fly ash containing lime and calcium chloride is quite slippery. To reduce the possibility of falls it is recommended that the area be cleaned after each individual sampling has occured. (Calcium chloride is hygroscopic and will become slippery on exposure to moisture in the air).

If the need arises to take bottom ash samples, personnel are cautioned to move ash discharge chutes away from their vicinity as engine block size lumps of hot waste may fall in their direction.

Sampling personnel must ensure that lock-out procedures at the main breaker panel are followed if attempting to sample from the flights of screw conveyors. It is not sufficient to only lock out at the local motor switch as these conveyors can often also be remotely started from the control room.

7) SAMPLING EQUIPMENT & SAFETY EQUIPMENT

The following is a suggested list of equipment that will be required in a sampling program.

- Hard Hats
- Approved safety boots
- Coveralls with long sleeves. (1 pair/person/day + spares)
- Gloves. Both cotton and rubber should be available (1 pair/person/day + spares)
- Dust Masks. Disposable or cartridge type (depending on plant regulations)
- Hearing protectors (insertion type).
- Eye protection. Safety glasses and goggles or full face shield
- Flashlights
- Tools: Shovels, wire cuters, tin snips, pliers, pipe wrenches, hammers, etc.
- Sample containers: Glass and/or PET wide mouth jars, 20 litre (5 gallon) pails, 200 litre (45 gallon) drums (detachable head) (the pails and drums should not be painted or otherwise coated on their interior)
- Ice packs when required
- Weigh scale of 200 kg (450 lb) capacity
- Steel strip for quartering sample, approximately 100 x 10 x 0.2 cm (3' x 4" x 1/8")
- Steel scoops
- Small brush
- Pipe thread tape (PTFE)
- Note books, pens, pencils, labels, adhesive tape, permanent markers for glass etc.
- Appropriate sample request forms etc.

RF/lb
TA 02 14 02
D7;RF,2228R

APPENDIX

ONTARIO MINISTRY OF THE ENVIRONMENT
STATISTICAL TEST FOR INCINERATOR ASH LEACHATE TOXICITY (EXAMPLE ONLY)

COMPLIANCE TESTING FOR LEAD

CONCENTRATION, (from Schedule 4, O.Reg.309)
= 0.05

sample 1	4.11
sample 2	3.13
sample 3	0.56
sample 4	2.19
sample 5	2.34
sample 6	1.14
sample 7	3.39
sample 8	2.12
sample 9	5.16
sample 10	4.34
sample 11	3.42
sample 12	1.35
sample 13	1.64
sample 14	0.74
sample 15	3.28
sample 16	1.07
sample 17	2.47
sample 18	
sample 19	
X =	2.48
s =	1.32
n =	17

Table 1:

(n-1)	t(.9)
1	3.078
2	1.886
3	1.638
4	1.533
5	1.476
6	1.440
7	1.415
8	1.397
9	1.383
10	1.372
11	1.363
12	1.356
13	1.350
14	1.345
15	1.341
16	1.337
17	1.333
18	1.330
19	1.328
20	1.325

From Table 1, for (n-1)
degrees of freedom, where
n = no. of samples: t = 1.337

u = 1.s/sqrt(n) = 0.427329

On the basis of the samples taken, the material as tested
is not leachate toxic

```
A1: 'ONTARIO MINISTRY OF THE ENVIRONMENT
A2: 'STATISTICAL TEST FOR INCINERATOR ASH LEACHATE TOXICITY
A4: '    COMPLIANCE TESTING FOR
A6: '    CONCENTRATION, (from Schedule 4, O.Reg.309)
C7: " =
H11: '     Table 1:
C12: 'sample 1
H12: "(n-1)
I12: '  t(.9)
C13: 'sample 2
H13: 1
I13: (F3) 3.078
C14: 'sample 3
H14: +H13+1
I14: (F3) 1.886
C15: 'sample 4
H15: +H14+1
I15: (F3) 1.638
C16: 'sample 5
H16: +H15+1
I16: (F3) 1.533
C17: 'sample 6
H17: +H16+1
I17: (F3) 1.476
C18: 'sample 7
H18: +H17+1
I18: (F3) 1.44
C19: 'sample 8
H19: +H18+1
I19: (F3) 1.415
C20: 'sample 9
H20: +H19+1
I20: (F3) 1.397
C21: 'sample 10
H21: +H20+1
I21: (F3) 1.383
C22: 'sample 11
H22: +H21+1
I22: (F3) 1.372
C23: 'sample 12
H23: +H22+1
I23: (F3) 1.363
C24: 'sample 13
H24: +H23+1
I24: (F3) 1.356
C25: 'sample 14
H25: +H24+1
I25: (F3) 1.35
C26: 'sample 15
H26: +H25+1
I26: (F3) 1.345
C27: 'sample 16
H27: +H26+1
```

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I27: (F3) 1.341
C28: 'sample 17
H28: +H27+1
I28: (F3) 1.337
C29: 'sample 18
H29: +H28+1
I29: (F3) 1.333
C30: 'sample 19
H30: +H29+1
I30: (F3) 1.33
H31: +H30+1
I31: (F3) 1.328
C32: "X =
D32: (F2) @AVG(D12..D30)
H32: +H31+1
I32: (F3) 1.325
C33: "s =
D33: (F2) @STD(D12..D30)
C34: "n =
D34: @COUNT(D12..D30)
A36: 'From Table I, for (n-1)
A37: 'degrees of freedom, where
A38: ' n = no. of samples:   t =
D38: (F3) @VLOOKUP((D34-1),H13..I32,1)
A40: "         u = t.s/sqrt(n) =
D40: +D38*D33/@SQRT(D34)
A44: 'On the basis of the samples taken, the material as tested
A45: "is
B45: @IF(D32+D40<100*D7,"not leachate toxic","leachate toxic")
```

ONTARIO MINISTRY OF THE ENVIRONMENT
STATISTICAL TEST FOR OUTLIER REJECTION (>10 SAMPLES) (EXAMPLE ONLY)

COMPLIANCE TESTING FOR LEAD

CONCENTRATION, (from Schedule 4, O.Reg.309)
= 0.05

sample 1	21.01
sample 2	5.16
sample 3	4.34
sample 4	4.11
sample 5	3.42
sample 6	3.39
sample 7	3.26
sample 8	3.13
sample 9	2.47
sample 10	2.34
sample 11	2.19
sample 12	2.12
sample 13	1.64
sample 14	1.14
sample 15	1.07
sample 16	1.05
sample 17	0.74
sample 18	0.56
sample 19	

Xn = 21.01
Xn-2 = 4.34
X3 = 1.05

Table II:

n	r
11	0.546
12	0.546
13	0.546
14	0.546
15	0.525
16	0.507
17	0.490
18	0.475
19	0.462
20	0.450
21	0.440
22	0.430
23	0.421
24	0.413
25	0.406

From Table II, where
n = no. of samples: r = 0.475

r22 = (Xn-Xn-2)/(Xn-X3) = 0.835

On the basis of the samples taken, the maximum value obtained should be rejected.

```
A1: 'ONTARIO MINISTRY OF THE ENVIRONMENT
A2: 'STATISTICAL TEST FOR OUTLIER REJECTION (>10 SAMPLES)
A4: '    COMPLIANCE TESTING FOR
A6: '    CONCENTRATION, (from Schedule 4, O.Reg.309)
C7: " =
H11: '     Table II:
C12: 'sample 1
H12: "n
I12: ^r
C13: 'sample 2
H13: 11
I13: (F3) 0.546
C14: 'sample 3
H14: +H13+1
I14: (F3) 0.546
C15: 'sample 4
H15: +H14+1
I15: (F3) 0.546
C16: 'sample 5
H16: +H15+1
I16: (F3) 0.546
C17: 'sample 6
H17: +H16+1
I17: (F3) 0.525
C18: 'sample 7
H18: +H17+1
I18: (F3) 0.507
C19: 'sample 8
H19: +H18+1
I19: (F3) 0.49
C20: 'sample 9
H20: +H19+1
I20: (F3) 0.475
C21: 'sample 10
H21: +H20+1
I21: (F3) 0.462
C22: 'sample 11
H22: +H21+1
I22: (F3) 0.45
C23: 'sample 12
H23: +H22+1
I23: (F3) 0.44
C24: 'sample 13
H24: +H23+1
I24: (F3) 0.43
C25: 'sample 14
H25: +H24+1
```

```
I25: (F3) 0.421
C26: 'sample 15
H26: +H25+1
I26: (F3) 0.413
C27: 'sample 16
H27: +H26+1
I27: (F3) 0.406
C28: 'sample 17
C29: 'sample 18
C30: 'sample 19
C32: "Xn =
D32: (F2) @MAX(D12..D30)
C33: "Xn-2 =
D33: (F2) +D14
C34: "X3 =
D34: ERR
A37: 'From Table II, where
A38: ' n = no. of samples:   r =
D38: (F3) @VLOOKUP((@COUNT(D12..D30)),H13..I34,1)
A40: " r22 = (Xn-Xn-2)/(Xn-X3) =
D40: (F3) (D32-D33)/(D32-D34)
A44: 'On the basis of the samples taken, the maximum value obtained
A45: "should
B45: @IF((D40>D38),"be rejected.","not be rejected.")
A47: !::
```